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Applicant	Serguei Vatchiants
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Title of Application:	A Method For Production Of Metal Foam Or Metal-Composite Bodies With Improved Impact, Thermal And Sound Absorption Properties
Confirmation No. 7762	Art Unit: 1742
Examiner	

Commissioner for Patents
Post Office Box 1450
Alexandria, VA 22313-1450

Submission of Priority Documents

Dear Sir:

Applicant hereby submits certified copies of the priority documents, Canadian Application No. 2,332,674 and Canadian Application No. 2,344,088 to perfect Applicant's claim of priority.

Respectfully submitted,

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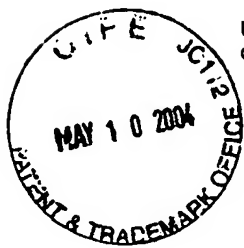


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Specification and Drawings, as originally filed, with Application for Patent Serial No:
2,332,674, on January 29, 2001, by **AGS TARON INVESTMENTS INC.**, for "A
Method for Production of a Foam Metal".

Tracy Pauline
Agent certificateur/Certifying Officer

July 3, 2003

Date

Canada

(CIPO 68)
04-09-02

OPIC  CIPO

A METHOD FOR PRODUCTION OF A FOAM METAL

FIELD OF THE INVENTION

The present invention relates to the field of powder metallurgy. More specifically, it concerns a method of manufacturing foamable metal bodies and their use.

5 BACKGROUND OF THE INVENTION

Already known in the prior art, there is German patent no 4101630 (US patent no 5,151,246) which describes a method for the production of porous semi-finished products from aluminum and copper-based alloy powders. The method described therein comprises the steps of mixing of an alloy powder with a foaming agent, filling
10 a press container with the mixture, simultaneously heating the filled container and applying pressure at which the foaming agent does not decompose, simultaneously cooling and removing the pressure, disassembling of the container followed by pushing of the solid briquette out of it, which is immediately heat treated to produce a porous body or is subjected to preliminary hot deformation via extrusion or rolling
15 followed by heat treatment. A very narrow range of products in terms of sizes and shapes can be produced with such method since the weight of the briquette is 2-5 kg. In addition, this method demonstrates a very low output because of the prolonged heating of the large size press container filled with the powder mixture. Even in the case where the powder mixture would be heated in a container having 100 mm in
20 diameter and 400 mm in height, the heating operation would be economically not feasible.

Also known, there is a method for the production of porous semi-finished products from metallic powders that incorporates different variants.

A first variant includes the steps of coating the bottom floor of a press container with
25 a metallic layer free of foaming agent, covering the metallic layer with a powder

mixture comprising a foaming agent, and then covering the layer of powder mixture with a second metallic layer free of foaming agent. The container is then heated, and hot compaction is carried out. This operation is the end of the method. The shape of the body produced can be changed via deformation. Then, the body is foamed for
5 formation of a new body wherein a high porous foamed metallic layer appears between two metallic layers.

A second variant includes the steps of disposing a dense metallic disk in an empty press container adapted for extrusion and filling the container with a powder mixture containing a foaming agent. Then, the container with the powder mixture is subjected
10 to heating followed by the application of a pressure of about 60 MPa. Due to the pressure, the central part of the hard metallic disk, which blocks the hole of the press die, begins flowing through this hole and ensures extrusion process. During subsequent extrusion stages, the compacted powder mixture plastically deforms and flows through the die hole also. In this case, the dense metallic layer covers the
15 extruded powder mixture which is ready for foaming. After foaming of this combined body the metallic layer covers a core consisting of a high porous foam.

The combined billets produced via both variants can be further rolled in sheets, and due to a heat treatment temperature, can be transformed in a porous metallic body (US Patent 5,151,246, September, 1992, B 22 F 3/18, B 22 F 3/24).

20 The disadvantage of this technique is also the limited possibility of production of semi-finished products, especially sheets of commercial sizes, low product yield and output, high manufacturing cost.

The method of the present invention is distinct from and overcomes several disadvantages of the prior art, as will be discussed in detail below.

SUMMARY OF THE INVENTION

An object of the present invention is to provide a method for the production of a foamable metal body that overcomes several of the above-mentioned drawbacks.

More particularly, the present invention proposes a method for the production of porous powder metallurgy metal products comprising the steps of:

- a) providing a mixture of metal alloy powders with a foaming agent;
- b) pouring the mixture of step a) in a mould, preferably a reusable mould;
- c) pre-compacting the mixture, preferably by vibration;
- d) heating the mould with the mixture at a temperature which ensures liquid-phase sintering of the mixture in order to produce a liquid phase sintered briquette;
- e) hot compaction of the sintered briquette to achieve a porous and brittle body where the foaming agent is embedded in a metal matrix;
- f) reducing the porous and brittle body into chips and continuously hot rolling the chips to form the desired semi-finished dense product;
- g) conventional deformation of the dense product to desired shape
- h) foaming of the shaped dense product by heating the same at a temperature sufficient to allow the foaming agent to decompose, and thereby forming the foamed end product.

According to a preferred feature of the invention, the hot rolled dense blanks are deformed by conventional methods to the desired shape by installing the same in a special mould, which cavity shape is in compliance with the shape of the end

product, and heated above a temperature of transition from solid to liquid state of an alloy to produce a porous net shape product.

Also preferably, prior to step h) of heating, two or more sheet blanks along any of their sides are put together with overlap to increase the length or width of the combined blank. These blanks are then heated above a temperature of transition from solid to liquid state of an alloy to form a porous product with doubled (tripled ...) width or length.

According to a preferred embodiment of the invention, the method incorporates mixing of powder aluminum alloys of various systems: Al-Cu-Mg-Si, Al-Mg-Si, Al-Mg-Cu-Si (cast alloys), Al-Cu-Mg-Mn, Al-Mg-Cu, Al-Zn-Cu-Mg, Al-Zn-Mg-Cu (wrought alloys) with a foaming agent having a decomposition temperature exceeding that of the solidus of the aluminum alloy powder matrix. The mixture obtained is filled in a split reusable mould which is heated with the powder mixture. Heating of the powder mixture is carried out at a temperature which ensures liquid-phase sintering after cooling to the temperature 10-20°C below the solidus of most fusible eutectics. As a result, the powder mixture loses its looseness. After removing the bottom of the mould, the hot mould is placed on the container of a vertical press. The ram of this press pushes the sintered powder mixture out of the mould into the press container, then a dummy-block is placed and hot compaction of the sintered powder mixture is carried out at a low specific pressure to produce porous (86-92 % relative density), and easy breakable briquettes. Using highly efficient machines, the cooled briquettes are reduced to fragment-shaped chips with powder particles of 0.5-5.0 mm in size, chemical composition of which conforms to that of initial aluminum alloy powder with uniform distribution of the foaming agent. Then the chips produced are heated with a high rate to a temperature below the solidus temperature of the lowest melting point eutectic by 10-20°C and is fed in the rolls of a powder rolling mill. Tangential stress action results in development of active shear strains in the chips in the deformation zone. Chips form new strong metallic bonds which sustain pronounced

tensile stresses during formation of a continuous dense hot-rolled sheet with required thickness. The hot-rolled sheets produced are cut to blanks which are fed to a heat treatment. High-temperature heat treatment is carried out by heating of the blanks from below on overheated melts of salts or metals above a temperature of phase transition from solid to liquid state and, based on the visual examination, foaming is stopped on the required height. The technical result obtained due to realization of the invention incorporates high labor productivity, creation of commercial waste-free production of porous semi-finished sheet.

The method of the present invention can be used for production of porous metal bodies for the civil-engineering machinery, automotive and aircraft and other industries wherein combination of such unique properties of this material as high specific strength and rigidity, energy absorption, heat insulation and sound-proofing, light weight, incombustibility, buoyancy and absolute environmental acceptability are required.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other objects and advantages of the invention will become apparent upon reading the detailed description and upon referring to the drawings in which figures 1 to 7 are schematic representations of the sequence of steps of a method according to a preferred embodiment of the invention. The detailed description of each figure is as follows:

Figure 1 schematically represents the step of mixing of metal powders with a gas propellant powder;

Figure 2 represents the step of pouring the mixture into a reusable mould and of precompacting the mixture by vibration;

Figure 3 represents the step of liquid phase sintering;

Figure 4 represents the step of pushing a liquid phase sintered briquette from a reusable can into press mould;

Figure 5 represents the step of compaction of the sintered briquette;

Figure 6 represents the step of reducing the body into chips, and continuously hot
5 rolling the chips into semi-finished dense product;

Figure 7 represents the step of foaming;

Figure 8 is a phase diagram of the powder alloy: Al-Zn-Cu-Mg; and

Figure 9 is a phase diagram of the powder alloy: Al-Mg-Cu-Mn.

While the invention will be described in conjunction with an example embodiment,
10 it will be understood that it is not intended to limit the scope of the invention to such embodiment. On the contrary, it is intended to cover all alternatives, modifications and equivalents as may be included as defined by the appended claims.

DESCRIPTION OF A PREFERRED EMBODIMENT

The purpose of the present invention is the production of complex shape products
15 out of continuous hot-rolled sheets of commercial sizes made from the chips produced from porous brittle hot-compacted briquettes. The technical result obtained due to realization of the invention incorporates a dramatic increase in product yield (creation of a waste-free technology), a reduction in manufacturing cost of porous products, a broadening of the range of products in terms of both their geometrical
20 sizes and density.

Referring to figures 1 to 7, the method of production of porous products from aluminum alloys incorporates mixing of an aluminum alloy powder (1) comprising

metal particles of an aluminum alloy selected from the group consisting of: Al-Cu-Mg-Si, Al-Mg-Si, Al-Mg-Cu-Si (cast alloys), Al-Cu-Mg-Mn, Al-Mg-Cu, Al-Zn-Cu-Mg, Al-Zn-Mg-Cu (wrought alloys) with a powder of a foaming agent (2), the foaming agent (2) having a decomposition temperature exceeding that of solidus of the aluminum alloy powder matrix. The mixture (5) obtained is filled in a split reusable mould (6) which is heated with the powder mixture (5), as shown in figure 3. Heating of the powder mixture (5) is carried out at a temperature which ensures liquid-phase sintering after cooling to 10-20°C below solidus temperature of the lowest melting point eutectic. As a result, the powder mixture now in the form of liquid phase sintered briquettes (12) loses its looseness. After disassembling of the mould (6), the hot mould is placed on the container (14) of a vertical press. The ram (13) of this press (14) pushes the sintered powder mixture (12) into the press container (14), then dummy-block is placed and hot compaction of the sintered powder mixture is carried out at a low specific pressure, as shown in figure 5. The hot-compacted briquettes (15) produced show a density of 86-92 rel. %. These briquettes (15) compacted at a low pressure are porous (8-14 rel. %) and brittle, thus easily breakable. Referring to figure 6, using highly efficient machines, the cooled briquettes (15) are reduced to fragment-shaped chips (18) with chips particles of 0.5-5.0 mm in size, chemical composition of which conforms to that of initial aluminum alloy powder with uniform distribution of the foaming agent. Then, the chips (18) produced are heated with a high rate to a temperature which is 10- 20°C below the solidus temperature of the low-melting point eutectic of the aluminum alloy. Then, the heated chips (18) are fed in the rolls (22) of a powder rolling mill. Tangential stress action results in development of active shear strains in chips in the deformation zone. Chips form new strong metallic bonds which sustain pronounced tensile stresses during formation of a continuous dense hot-rolled sheet with required thickness. The hot-rolled sheets (23) produced are cut to blanks which are fed to a heat treatment . Referring to figure 7, high-temperature heat treatment is carried out by heating of the blanks (24) from below on overheated melts of salts or metals (27) at a temperature above a

temperature of phase transition from solid to liquid state and, based on the visual examination, foaming is stopped on the required height.

According to a preferred aspect of the invention, the hot-rolled sheet blanks are subjected to a high-temperature heat treatment via heating of the blanks above a
5 temperature of phase transition from solid to liquid state and the foaming process is stopped by a plate fixed at a required height when the foaming process is examined visually. Alternatively, the foaming process is stopped by a plate with the required technological projections on its internal surface and fixed at the required height when the foaming process is examined visually.

- 10 According to another preferred aspect of the invention, the hot-rolled sheet blanks are used to form a composite block comprising a lower metallic sheet with a special coating, the hot-rolled blank placed on the surface of the lower metallic sheet, and an upper metallic sheet with the same coating is located above the hot rolled blank at a certain height. Connecting cross elements are provided to ensure bracing of the
15 upper and lower metallic sheets. The cross elements simultaneously play a role of fastening and connecting elements. For foaming the product, the composite block is heated from below in a furnace up to a temperature of phase transition from solid to liquid state. The foaming is watched visually and when the foaming aluminum blank reaches the upper second metallic sheet, the composite block is removed out
20 of the furnace and cooled.

Preferably, the upper metallic sheet is a volumetric sheet stamping of a desired shape.

- Also preferably, the chips (18) are classified by grain sizes from 1.5 up to 5.0 mm, each size fraction is mixed with fine refractory material powders passive to aluminum,
25 then the mixture is filled in moulds and heated in a furnace up to a foaming temperature which exceeds the transition temperature from solid to liquid state by

50-70°C; after completion of the foaming process, the mixture is screened to separate the refractory material powders from porous granules.

According to another preferred embodiment of the invention, the chips produced are classified by grain sizes from 1.5 up to 5.0 mm, each size fraction is heated up to a temperature below the solidus point of the alloy by 10-100°C and then dispersed as a monolayer on a flat heated surface and then the fragment-shaped chips are pelletized by circular movements of a heated massive disk-shaped plate; then each fraction of the pellets produced is mixed with fine refractory material powders passive to aluminum and then the mixture is filled in moulds and heated up to a foaming temperature which exceeds transition temperature from solid to liquid state by 50-70°C; after completion of the foaming process the mixture is screened to separate spherical porous granules from the fine refractory material powder.

According to a further preferred embodiment, the chips produced are classified by grain sizes from 1.5 up to 5.0 mm, each size fraction is dispersed as a monolayer on a special base, heated from below up to a temperature of phase transition to liquid state; when it is examined visually that the foaming particles reach the desired size, the foamed granules are removed out of the furnace.

The foamed granules may then preferably be mixed with a resin and injected into the internal space of any structural element comprising one or more hollow pieces. The resin is cured to increase stiffness and energy absorption of the structural element.

According to another aspect of the invention, the chips which are not screened to size fraction are used to form a composite block which contains a flat metallic sheet with special coating on the surface of which a layer of chips is dispersed and, above this layer, at a certain height, the second metallic sheet with special coating, stamped beforehand for the desired component, is located and after this, the composite block formed is heated in a furnace up to a foaming temperature which exceeds the transition from solid to liquid state by 50-70°C and when it is examined visually that

the foaming particles reach the upper metallic layer, the block with foamed powders is removed out of the furnace and cooled. (To provide for heating of the block in inert atmosphere).

5 Preferably in this case, to ensure bracing between the sheets, they are fastened together by connecting cross-pieces which simultaneously play a role of fastening connecting elements.

Also preferably, the chips which are not screened to size fraction can be used to fill to the desired volume fraction the internal space of any structural element comprising one or more hollow pieces. The whole assembly is heated above a temperature of transition from solid to liquid state of an alloy to form porous filler (core).
10

According to a further aspect of the invention, the rolling of the heated chips is conducted together and between two or more heated metal sheets to produce a composite body. The produced composite body is heated above a temperature of transition from solid to liquid state of an alloy to form a multilayer structure with porous core and metallic bonds between core and facings.
15

The possibility of realization of the invention characterized by the above-mentioned set of the signs and the possibility of the realization of the purpose of the invention can be corroborated by the description of the following examples.

Example 1

20 The example of the realization of the method for production of flat porous semi-finished products is as follows.

Al-Mg-Cu-Mn aluminum alloy powder (a liquidus temperature of the alloy is 640-645°C, a temperature of low-melting point eutectic is 505°C) of 300 kg in weight was mixed with TiH₂ foaming agent of 3.25 kg in weight (a decomposition temperature

is 690°C and filled in a split mold of 340 mm diameter, 800 mm in height with internal space of 290 mm in diameter. Figure 8 shows a vertical cross section of the phase diagram of Al-Mg-Cu-Mn alloys. Hatched zone in this figure represents alloys used in the process. As can be appreciated, the average solidus temperature is 503°C and the liquidus temperature is approximately 650°C. The powder mixture was compacted by vibration to obtain a density of 1.75-1.8 g/cm³. Weight of the mixture in each mould was from 97 up to 100 kg. The powder mixture was heated at a temperature of 510-515°C to ensure liquid-phase sintering after cooling down to a temperature of 480-485°C, the powder mixture lost its looseness. After disassembling of the mould, the hot mould was placed on the container of a 10 MN or 15 MN capacity vertical press. The diameter and height of the container were 300 and 800 mm respectively. The ram of the press pushed the sintered powder mixture into the press container, then a dummy-block was placed and hot compaction of the sintered powder mixture was carried out at a low specific pressure of 140-200 MPa. The hot-compacted briquettes produced showed a density of 86-92 rel. %. After cooling the briquettes were reduced on special machines to fragment-shaped chips. Then the chips produced was heated at a high rate in a furnace and fed on a powder rolling mill on which 290 kg of sheet was continuously rolled. The hot-rolled sheet was cut to 1000 x 1200 x 5 mm blanks using shears installed behind the rolling mill. The first part of the blanks was used for free foaming without clamping from above. High-temperature heat treatment was carried out by heating of the sheet blanks from below on overheated melts of salts. Based on the visual examination, the foaming process was stopped by quick removal of the foamed sheet from the furnace when thickness was 24.5 mm. The size of the porous semi-finished products was 1005 x 1210 x 24.5 mm. The lower surface of the porous sheets was smooth, while the upper surface was smooth also but had traces of bulgings appeared due to internal pores. The density of the porous semi-finished products produced was 0.56-0.58 g/cm³. Porous semi-finished product yield was 95%.

The second part of the sheet blanks was foamed in the limited space in terms of height. Heat treatment of the sheet blanks was carried out from below at a temperature of overheated salt melt. When a thickness of 24.5 mm was obtained, the foaming process was stopped by a plate fixed at this height. The completion of
5 the foaming process was determined visually also. The foamed block was quickly removed out of the furnace. The density of the porous semi-finished products produced was 0.56 g/cm³. The upper surface of the porous sheets was dead and smooth. Porous semi-finished product yield was 95-97%.

Scrap was insignificant, in the form of side crops formed after trimming of the hot-
10 rolled sheets. The production process is developed by such a way, that scrap is almost absent and it is reduced into chips and brought back into hot-rolling process. Heating of the primary powders above a temperature of appearance of low-melting point eutectic by 10-20°C and subsequent cooling below this temperature by 20-30°C ensure development of liquid-phase powder sintering. The powder mixture in this
15 state loses its looseness and can be easily pushed from the mould into the press container. The first source of appearance of extremely low hydrogen amounts is decomposition of TiH₂ at a heating temperature. The second source is surface hydrogen appeared due to reaction of sorbate (H₂O molecules) with aluminum cations which diffuse through an oxide film. Surface hydrogen and hydrogen formed
20 due to decomposition of TiH₂ leave the porous briquettes partially, while the largest hydrogen amount is capable of dissolving in appeared low-melting point eutectic.

Then, hot compaction operation at a low pressure of 140 or 200 MPa is carried out. Pressure applied to a sintered briquette is able to form only a porous briquette. The porous state is necessary only to facilitate production of the chips on special
25 machines as these chips are the main initial material for the present invention. It is clear that the main operation i.e. hot compaction is a waste-free process in reality.

Heating and feeding of the heated chips on a powder rolling mill are almost waste-free operations also, while insignificant amounts of torn side edges formed during hot rolling shall be trimmed after the rolling and recycled in the process. Mastered technological process for foaming of dense hot-rolled sheets results in high product yield. Therefore, the yield of 95% measures up to real value.

The effect of the heating temperature on the chips should be specially discussed. The heating temperature of the chips prior to rolling preferably complies with hard high plastic state, that is to say, it should preferably be below a temperature of low-melting point eutectic formation by 40-60°C. When the heated chips is fed on a powder rolling mill at a temperature at which liquid phase, low-melting point eutectic is absent, chips are subjected to sequentially followed actions of the force field. At first, intensive compaction of the hot chips is carried out. The compacted chips which move to deformation zone come to the force field which works between rolls and causes appearance of tangential stresses which result in development of shear strains which destroy initial particles and form new contacts between new juvenile surfaces. Combination of a pressure and temperature ensures formation of strong metallic bonds on new interaction contacts between particles due to active diffusion. The formed strong metallic bonds are capable of sustaining noticeable tensile stresses in the deformation zone. As a result, when process parameters are chosen correctly, dense continuous sheets from 1.5 up to 6.0 mm in thickness can be formed.

Heating of the chips prior to rolling at a temperature above that of the appearance of low-melting point eutectic by 10-20°C results in the formation of the liquid phase. The liquid phase works as a lubricant in the zone of active deformation and appearing tangential stresses and does not result in the appearance of shear strains. In the zone of active deformation, wherein extra rise of a temperature due to friction forces is observed, the liquid phase in the deformation zone is the weakest point in the structure of a forming sheet. Tensile and tangential stresses destroy the sheet

continuum which is not formed. A hot-rolled sheet demonstrates cracks on the side edges, separation of sheet pieces, and in some cases, failure of the sheet takes place. The main cause of powder rolling process instability of this kind is formation of the liquid phase in chips particles during heating prior to rolling.

- 5 If the heating of the primary powder mixture is performed at a temperature of 10-20°C, which is below that of low-melting point eutectic formation, the sintered powder mixture obtained will retain its looseness. Transportation of the disassembled mould to the press container will be impossible, a briquette structure will be loose.

Example 2

- 10 The example of realization of the method for production of porous semi-finished products from the chips is as follows:

Al-Zn-Cu-Mg aluminum alloys powder (a liquidus temperature of the alloy is 630-640°C, a temperature of low-melting point eutectic formation is 480°C of 210 kg in weight was mixed with CaCO₃ foaming agent of 12 kg in weight (a decomposition

- 15 temperature is 720°C) and filled in a split mold of 340 mm in diameter, 800 mm in height with internal space of 290 mm in diameter. Figure 9 shows surfaces of crystallization (surfaces of liquidus) of the powder alloy Al-Zn-Cu-Mg containing Zn-4,5%, Cu 3,5-4,5%, Mg –negligible, Al-balance. The alloys used are in the AL corner of the diagram (small hatched zone) and have a liquidus temperature of 650°C.

- 20 Solidus of these alloys is in the interval of temperatures of 510-520°C. The powder mixture was compacted by vibration to obtain a density of 1.75-1.8 g/cm³. Weight of the mixture in each mould was from 97 up to 100 kg. The powder mixture was heated at a temperature of 490-500°C to ensure liquid-phase sintering after cooling down to 450-460°C and the mixture lost its looseness. After disassembling of the
- 25 mould, the hot mould was placed on the container of a 10 MN or 15 MN capacity vertical press. The diameter and height of the container were 300 and 800 mm

respectively. The ram of the press pushed the sintered powder mixture into the press container, then a dummy-block was placed and hot compaction of the sintered powder mixture was carried out at a low specific pressure of 140-200 MPa. The hot-compacted briquettes produced showed a density of 86-92 rel.%. After cooling, the
5 briquettes were reduced on special machines to fragment-shaped chips.

To realize the method, the chips produced were graded into grain sizes of 2.0, 3.0, 4.0 and 5.0 mm, each size fraction was mixed with fine refractory material powders passive to aluminum, the mixture was filled in moulds, heated in a furnace at a foaming temperature which exceeds the transition temperature from solid to liquid
10 state by 50-70°C; after completion of the foaming process, the mixture was screened to separate the refractory material powders from porous granules. The porous granules from 3.0 up to 10.0 mm in size and 0.3 up to 0.9 g/cm³ in density are a good filling agent for any shape of cases for energy absorbing components used in the automotive industry.

15 An easier technique for realization of the chips of the same alloy, graded into grain sizes of 2.0, 3.0, 4.0 and 5.0 mm is discussed below. Each fraction was dispersed as a monolayer on a special base, heated in a furnace from below on overheated melt of salt up to a foaming temperature; when it was examined visually that the foamed granules reached the desired size, they were removed out of the furnace
20 and cooled. The granules had a hemispheric shape with radius from 5.0 up to 20.0 mm and a density from 0.4 up to 1.0 g/cm³.

Porous granules of this size and shape can find application for production of volumetric noise suppression and fire barrier components and also large-size shock absorption elements. Product yield is 100%.

25 Although preferred embodiments of the present invention have been described in detail herein and illustrated in the accompanying drawings, it is to be understood that the invention is not limited to these precise embodiments and that various changes

and modifications may be effected therein without departing from the scope or spirit of the present invention.

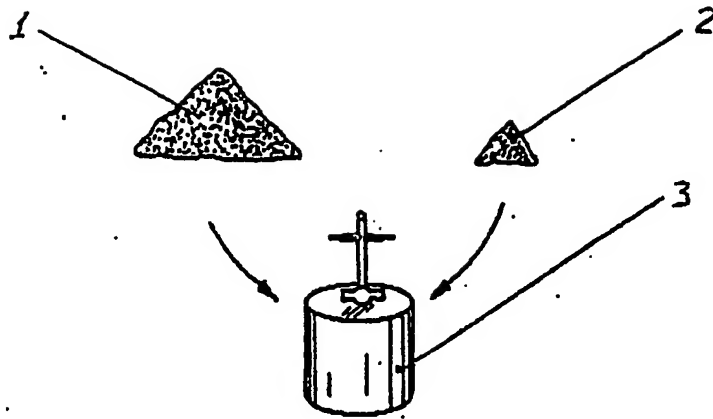


Fig. 1

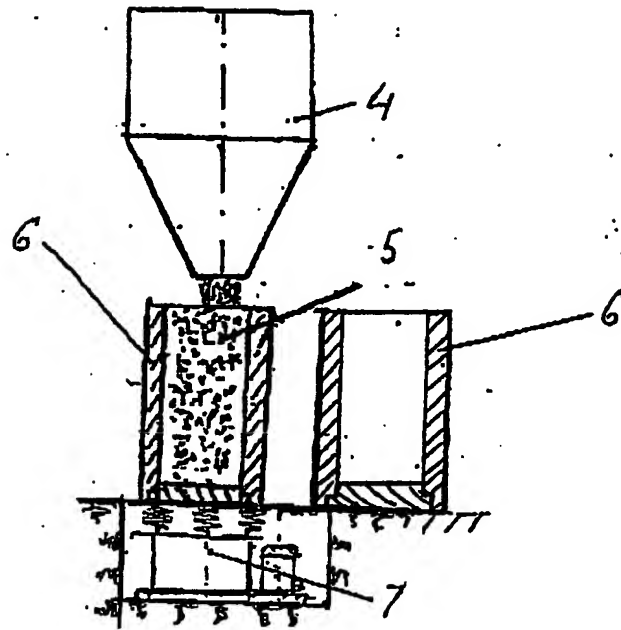


Fig 2.

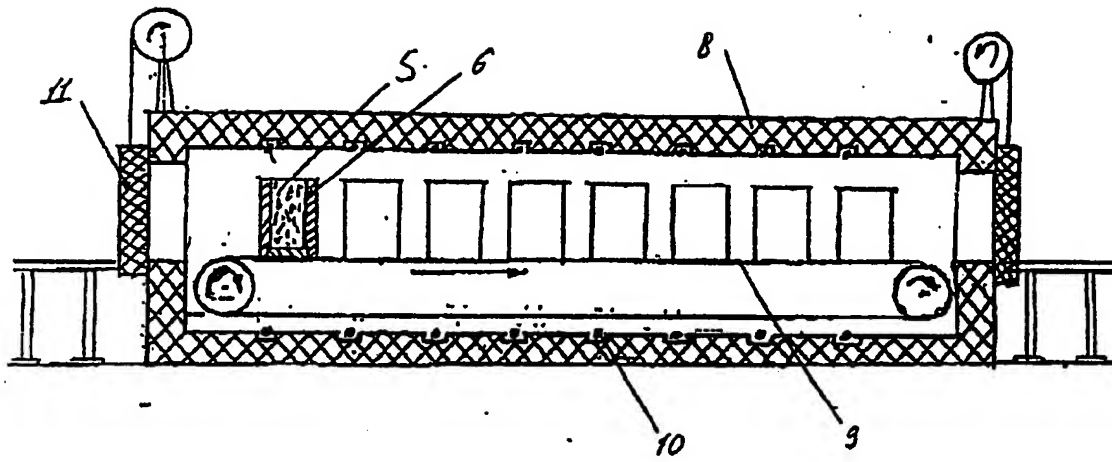


Fig. 3

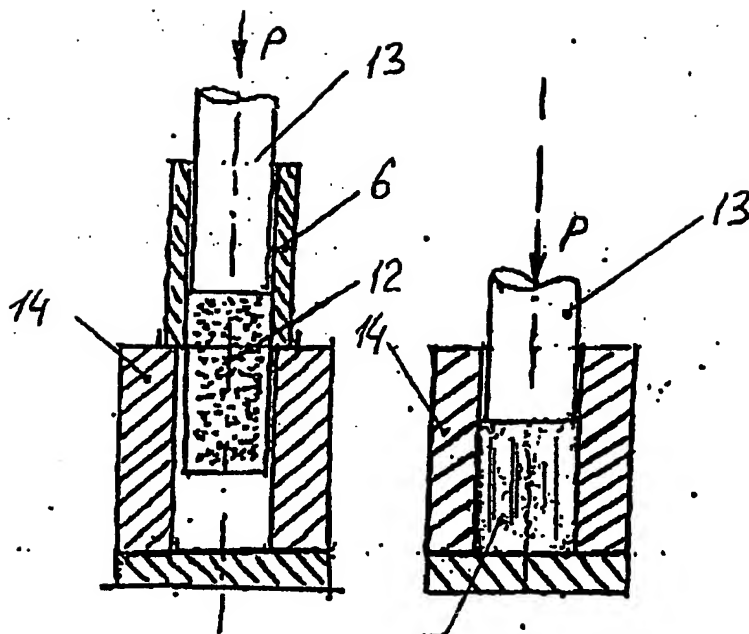
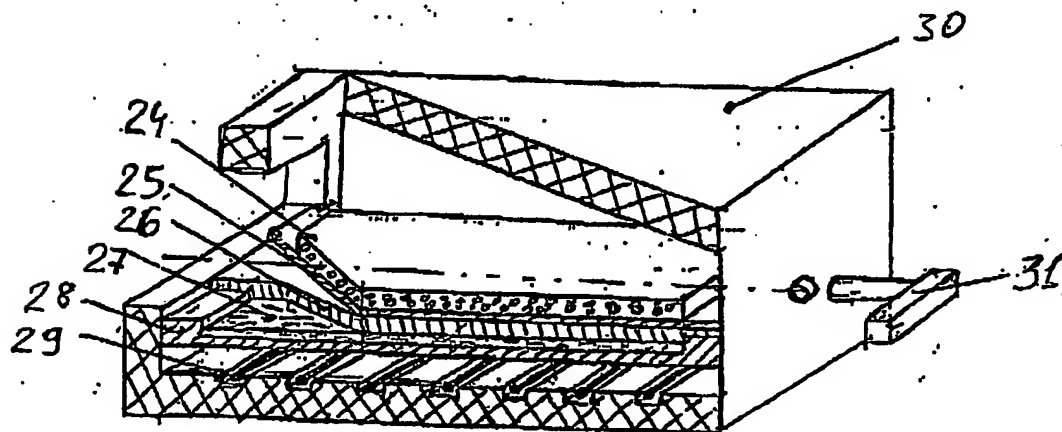
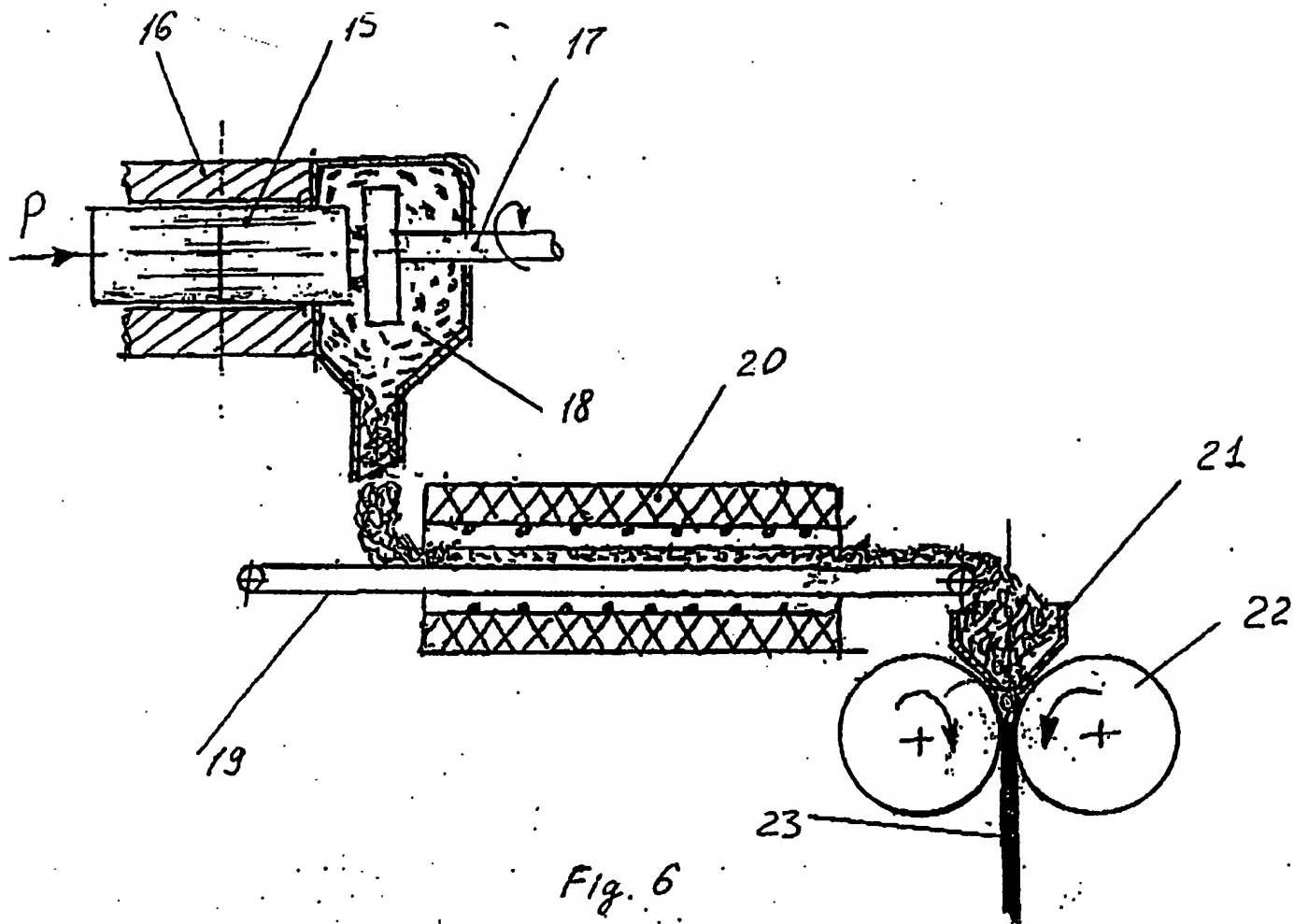


Fig. 4

Fig. 5



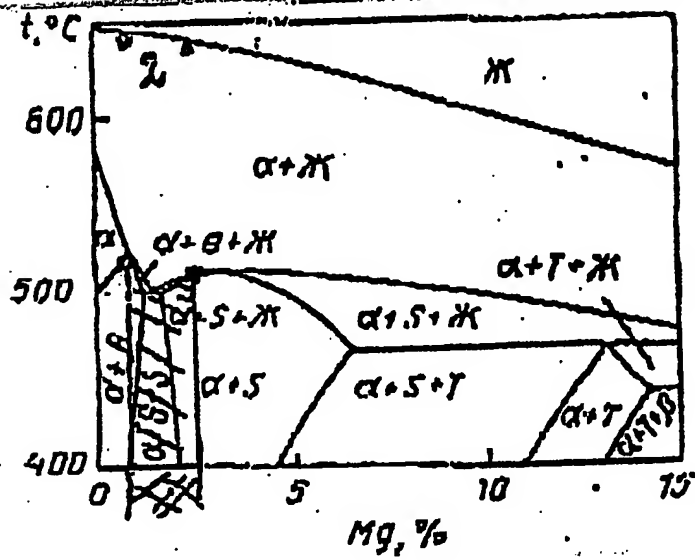


Fig. 8

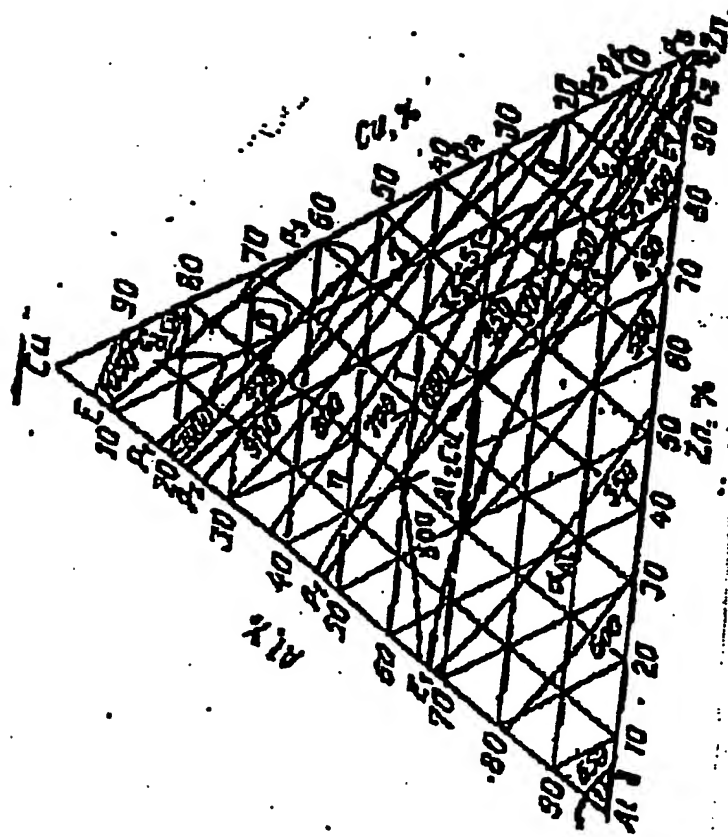


Fig 9